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Jones

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DECLARATION UNDER 37 C.F.R. 1.132

Dear Sir:

1. I, Samantha Jones, hereby declare and state the following:
2. I am a citizen of the United Kingdom. I reside at 121 Upper Ettingshall Road, Coseley, Bilston, West Midlands, UK.
3. I have a degree in Material Science from the University of Bath, received in 1988, and a Ph. D. from the University of Birmingham, received in 1992. I have been continuously employed by the University of Birmingham since September 1992, in the

School of Metallurgy and Materials, working in the field of investment casting research. My current position is Research Fellow, working on research and development of gamma titanium aluminide alloy investment casting.

4. I am the sole inventor of the subject matter of the above-identified patent application.

5. As a person having ordinary skill in the art of investment casting, I have reviewed United States Patent No. 3,894,572, to Moore, Jr., hereinafter "Moore." It is my opinion Moore does not disclose or suggest each and every feature of the claimed invention.

6. Moore does not disclose gel-forming materials that are superabsorbent polymers, which are required in the claims of the present application. There is no mention of these polymers anywhere within the reference.

7. The article attached in Exhibit 1 of this declaration, "Superabsorbent Polymers: An Idea Whose Time Has Come," by Fredric L. Buchholz, was printed in the Journal of Chemical Education, and is dated from 1996. This journal is an educational text.

8. As stated in the opening paragraph of the Buchholz article, superabsorbent polymers are cross-linked polyelectrolytes that, due to their ionic nature and interconnected structure, absorb large quantities of water and other aqueous solutions without dissolving. As stated at the bottom of the first column of p. 512, the polymer forms a gel, and holds the moisture in a "solid, rubbery state." The liquid is thus held within the polymer structure, and is prevented from leaking.

9. Moore does not mention at all that the polymers used for the chemical setting agents discussed therein show any degree of cross-linking, and therefore does not disclose superabsorbent polymers. The only discussion of cross-linking anywhere in

Moore is between the colloidal particles, and not within the setting agent polymers themselves. (col. 8, l. 34-41) From this, it is clear that any cross-linking that occurs in Moore is a result of the reaction between the setting agent and the colloidal particles. This is in contrast to the superabsorbent polymer of the present claims, which has a cross-linked structure within the polymer itself.

10. One of ordinary skill in the art would have no reason to believe that Moore discloses superabsorbent polymers, i.e. polymers that are cross-linked. The polymers of Moore are disclosed as "polyfunctional organic acids." These polymers are chosen because of their polyanionic characteristics, and high anionic density. These polyanionic polymers react strongly with the positively charged colloidal particles, which enhances the chemical setting process so important to Moore. (col. 8, l. 34 – 41)

11. Cross-linked polymers, by contrast, such as the superabsorbent polymers of the present application, provide no additional anionic function. They would therefore be of no use to Moore, since the process disclosed therein is so dependent on the interaction between the anionic groups of the polymer and the colloidal particles. Moore would therefore provide no guidance for one looking to develop the process of the present application, which requires an absorption of moisture by the gel-forming superabsorbent polymer.

I hereby declare that all statements made herein of my own knowledge are true and all statements made on information and beliefs are believed to be true. I further declare that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001, that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Dated: 2/4/09

By: S. Jones

Dr Samantha Jones
RESEARCH FELLOW

EXHIBIT 1

products of chemistry

edited by
GEORGE B. KAUFFMAN
California State University, Fresno
Fresno, CA 93740

Superabsorbent Polymers

An Idea Whose Time Has Come

Fredric L. Buchholz

The Dow Chemical Company, 1603 Building, Midland, MI 48674

A Brief History of Superabsorbents

Superabsorbent polymers are cross-linked polyelectrolytes. Because of their ionic nature and interconnected structure, they absorb large quantities of water and other aqueous solutions without dissolving. This makes them ideally suited as absorbents of body fluids in many personal care products sold today, including baby diapers, adult incontinence products and feminine napkins. But it wasn't always so. The widespread use of superabsorbent polymers in personal care products is a recent phenomenon, even though the basic materials and ideas have been around for some time. It's a perfect example of an idea whose time has finally come.

The commercially important superabsorbent polymers are sodium salts of cross-linked poly(acrylic acid), and graft copolymers of cross-linked poly(acrylic acid) and starch. Cross-linked and swellable poly(acrylic acid) was described by W. Kern in 1938 (1). He made it by thermally polymerizing an aqueous solution of acrylic acid and divinylbenzene. The synthesis, properties and physical chemistry of cross-linked poly(acrylic acid) and a very similar material, cross-linked poly(methacrylic acid) were studied in depth by Kuhn, Katchalsky and co-workers in the early 1950s (2, 3). A first practical use of cross-linked potassium polyacrylate, made by irradiating an aqueous solution of the monomer, was as a water immobilizing agent in fire-fighting, as described by Bashaw and Harper in a 1966 patent (4). And in 1968, Harper and co-workers claimed cross-linked polyacrylates to be useful as absorbents in diapers (5). In other patents, Harper and coworkers (6), and Harmon (7), claimed the use of similar materials in absorbent medical and personal care products in the United States. Nevertheless, the large scale application of these materials languished until the early 1980's, when diapers containing superabsorbent polymer were made and sold commercially in Japan (8). The products were a great success in Japan, and the idea was brought back to the United States by 1984. The new superabsorbent diapers were then marketed in Europe by the late 1980's. As a result, in just over 10 years the superabsorbent polymer industry grew to about 414,000 metric tons per year (9).

Uses of Superabsorbents

In baby diapers, superabsorbent polymers absorb, and retain under slight mechanical pressure, about 30 times their weight in urine. The resulting gel holds the liquid in a solid, rubbery state and prevents the liquid from leaking back onto the baby's skin and clothing. In a diaper, the polymer is mixed with 1–10 times its mass of cellulose fluff (wood fiber) to make up the absorbent core of the diaper.

The core is sandwiched between the porous top-sheet and the impermeable back-sheet to make a diaper (10). Before superabsorbent polymers, liquid in the absorbent core was held only in the open space between the fibers of cellulose fluff. In order to hold a large volume of liquid, the core had to have lots of open space and was, therefore, thick. Cores containing superabsorbents can be thinner because a small volume of dry superabsorbent polymer can absorb many times its volume of liquid. In addition to thinner infant diapers, thin, garment-like training diapers that contain superabsorbent polymer have become very popular. Superabsorbent polymers are also used in some feminine napkins and adult incontinence products.

Several applications are developing outside of the personal care industry, and for many of them, dealing with leaking water is also a common theme. For example, leaking water is often a problem in the construction industry. One new product is a sealing composite that swells slightly in water. It is made by blending a superabsorbent powder into rubber with the aid of a surfactant-like substance (11). The sealing composite is used, like mortar, between the concrete blocks of the building. If water contacts the composite, it swells to make a tight, impermeable barrier to further penetration of water. Such a composite was used in construction of the Channel Tunnel between France and England (12).

Leaking water can also degrade the performance of fiber-optic communication cables and power transmission cables. Water-blocking tapes prevent intrusion of water into the cables (13). The flexible, water-blocking tape is made by applying a superabsorbent polymer and a polymeric binder onto a non-woven fabric (14). The tape is wrapped around the cable, beneath the plastic covering, and intercepts any water that gets through it. Because the cables are exposed to seawater or ground water that contains di-valent cations, superabsorbents containing sulfonate functional groups, rather than carboxylic acid groups, are preferred for this application (15).

In addition to its liquid-water absorption characteristic, superabsorbent polymers absorb water from the vapor state and therefore can be used to control humidity. In fact, superabsorbents absorb and release moisture from the air more effectively than silica gel. This property can be used, for example, to prevent damage due to moisture condensing on walls and ceilings in humid buildings. Because powdered superabsorbents are difficult to apply in such cases, superabsorbent sheets or superabsorbent fibers have been developed. For some uses, such as absorbent pads in meat and poultry packages (16), the polymer is laminated between two layers of tissue. More technically-sophisticated

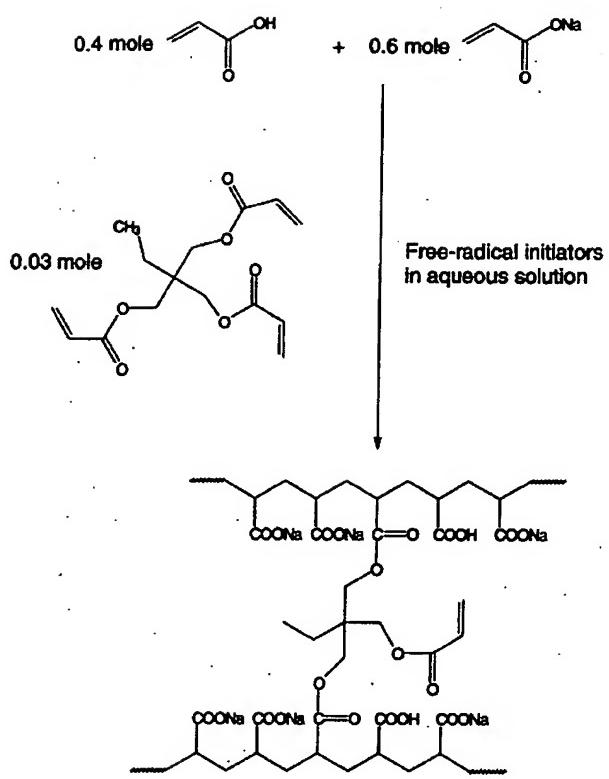


Figure 1. Free-radical polymerization of partially neutralized acrylic acid, and a cross-linker, in aqueous solution to form a superabsorbent polyacrylate.

sheets are made by polymerizing the monomers directly onto non-woven fabrics (17). These products help maintain a constant humidity in vegetable and fruit storage buildings and prevent spotting of the produce caused by water dripping from the surfaces of the structure (12).

Superabsorbents can also help conserve water in agriculture and horticulture. For this purpose, the polymers are mixed into soil at a concentration of about 0.1 wt%. This soil retains more moisture for longer times and plants live longer after germination. In one experiment, the yields of Chinese cabbage increased about 10 % compared to the control plot without superabsorbent polymer, using the same amount of water (12). In clay soils, superabsorbent polymer helps improve the air content of the soil.

Chemistry of Superabsorbent Polymers

In the manufacturing process of superabsorbent polymer, partially neutralized acrylic acid and a crosslinker, such as trimethylolpropanetriacrylate, react in aqueous solution by a free-radical chain addition mechanism (Fig. 1). The initiating radicals typically form by thermal homolysis of persulfate ions or by reaction of persulfate with a reducing agent such as sodium thiosulfate. The tough, rubbery reaction product is cut into small pieces and then dried in large, continuous ovens and the dry polymer cakes are milled into irregular granules (about 300500 micron mean particle diameter) that are used in diapers. The commercially available superabsorbents are manufactured by means of fairly complicated processes in order to tailor-make the materials for the desired applications. Literally hundreds of patents describe the details of the several processes employed, and extol the virtues of novel syn-

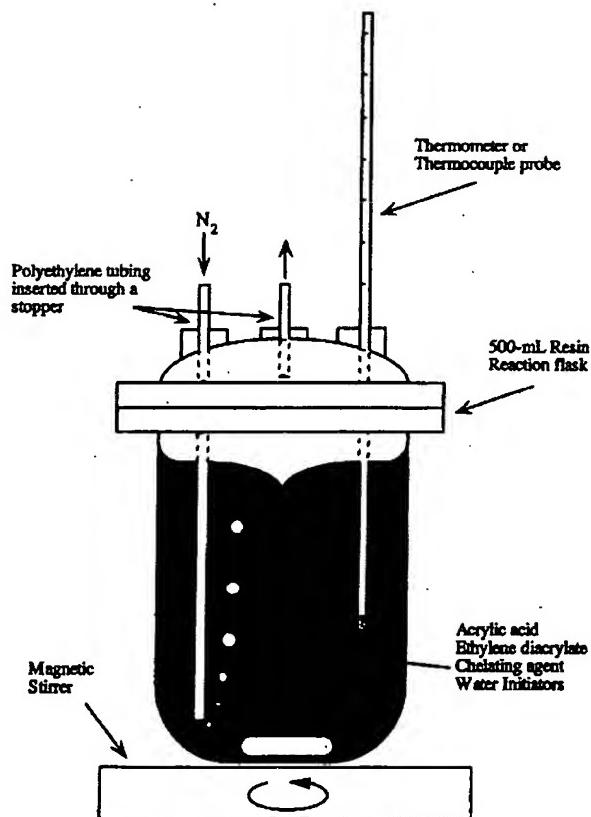


Figure 2. Simple polymerization reactor for superabsorbent polymer.

thetic steps. But a simply made polymer can suffice to demonstrate the properties and behavior of these fascinating substances.

A Simple Synthesis of Polyacrylate Superabsorbent Polymer

- Caution: Acrylic acid is an irritant. It is corrosive to skin. It is toxic by inhalation. It may polymerize explosively.

Assemble a polymerization reactor from a 500-mL resin reaction kettle, stoppers, polyethylene tubing, and a thermometer, as shown in Figure 2. The reactor contents may be stirred by means of a magnetic stirrer (1.25-in. PTFE-coated stir bar). Mix to prepare a monomer solution:

- 100.0 g acrylic acid [79-10-7]
- 300.0 g distilled water
- 0.10 g ethylenediaminetetraacetic acid [60-00-4]
- 1.00 g ethylene diacrylate [2274-11-5].

In contrast to most commercial processes that partially neutralize the acrylic acid before polymerization, here the monomer will be polymerized in its unneutralized form to afford a more brittle reaction product that is easily comminuted by hand. We shall also use the simple cross-linker ethylene diacrylate in place of trimethylolpropanetriacrylate (a complex mixture of esters) that is often employed commercially. Place the monomer solution into the reactor, then stir, and de-oxygenate with nitrogen gas, bubbling through the solution at a flow rate of 400 mL/min for 30 min. While the monomer solution is being de-oxygenated, make the initiator stock solutions as follows:

- Dissolve 0.5 g sodium persulfate [7775-27-1] in 4.50 g distilled water.
- Dissolve 0.5 g sodium thiosulfate pentahydrate [10102-17-7] in 4.50 g distilled water.
- Dissolve 50 mg tertiary butyl hydroperoxide [75-91-2] in 5.0 g distilled water.

When the requisite de-oxygenation time has passed, add the following amounts of the initiator solutions to the monomer solution, using pipets or syringes, in the order listed, with stirring:

- First, add 3.00 mL of sodium persulfate solution.
- Second, add 1.50 mL of tert-butyl hydroperoxide solution.
- Third, add 4.00 mL of sodium thiosulfate solution.

An exothermic reaction (*caution!*) begins within a few minutes if the monomer solution was adequately de-oxygenated. The temperature will rise by about 70 °C. The conversion of monomer may be followed by monitoring the fractional temperature rise if the resin reaction kettle is replaced by an adequately sized Dewar flask, fitted with a large stopper having small holes bored into it through which the nitrogen bubbling tube may be passed, and a thermometer inserted. The system becomes an adiabatic system whose kinetics may be followed in a straightforward way (18).

After the reaction has subsided, as evidenced by a drop in temperature, cool the reactor and its contents by immersing it in ice water. Slide the lump of clear, rubbery gel from the reactor by wetting a laboratory spatula with water, inserting it between the glass wall of the reactor and the gel, and sliding the spatula around the perimeter of the gel to free it from the glass. The lump of gel may be crushed by hand, using laboratory rubber gloves as skin protection (the acidic gel sticks tenaciously to bare skin). Then neutralize the crumbled mass of gel by adding a solution of 75.00 g sodium carbonate dissolved in 225.0 g of distilled water. Mix the carbonate solution and the crumbled gel together in a 4-L beaker, using a spatula. The neutralization proceeds with a snapping and hissing action as the carbon dioxide, which is liberated during neutralization, escapes from the elastic gel. Set the mixture aside for about an hour to assure complete absorption of the neutralizing solution. Dry the neutralized, superabsorbent polymer gel in a 100 °C oven. Crush the resulting glassy polymer in a mortar and pestle to obtain a granular material.

Analysis of Superabsorbents

The key properties of superabsorbent polymer are the swelling capacity and the elastic modulus of the swollen, cross-linked gel. Both of these properties depend upon the cross-link density of the network: modulus increases and swelling capacity decreases with increasing cross-link density. The cross-link density of superabsorbent polymer used in diapers is about 0.05 mole of cross-linker per liter of dry polymer. Because of this very low concentration, conventional spectroscopic techniques, such as infrared spectroscopy and nuclear magnetic resonance spectrometry, are not very useful for characterizing the cross-link density. The cross-link density can be estimated from measurements of the modulus or swelling capacity by applying an appropriate theoretical model. The modulus is measured by means of rheological methods and the swelling capacity is measured using gravimetric methods.

The most common measure of the swelling capacity is the centrifuged capacity (19). A porous bag, measuring about 3 inches square, is made from a heat-sealable, water-wettable fabric (a tea bag). A few hundred milligrams of the granular polymer is put into the bag, the bag is sealed, and then it is immersed in the test fluid, which is usually a solution of 0.9% NaCl in water, and left to swell

for 30–90 min. The immersion time depends on the particle size of the granules and their swelling rate. The bags are withdrawn from the bath and placed into a laboratory centrifuge, equipped with a perforated spin-basket, and centrifuged for a few minutes to remove any unabsorbed fluid from between the particles of the gel. Alternatively, the wet bag may be blotted with a paper towel. The swelling capacity is calculated from the ratio of the mass of fluid absorbed to the mass of dry polymer.

The elastic shear modulus of swollen gels is typically measured by means of an oscillatory stress rheometer (20). The swollen mass of particles is packed between the plates of the rheometer, and an oscillating shear stress is applied to one plate. The motion of the opposite plate is measured and depends on the damping of the mechanical wave passing through the sample. The shear modulus is calculated from the measurements.

Other tests are used to measure fluid absorption under conditions that simulate those in use. For example, the polymer in a diaper is under compression when a baby sits on the diaper. The absorbency under load test (21) measures the swelling capacity of the polymer while an external pressure is applied to the swelling gel. Similar information may be collected by means of measurements of the swelling pressure, which is the pressure generated by the gel in contact with an external source of fluid while being confined to a given volume (22). These tests are related to the osmotic compressibility of the network, which is a measure of how easily a given gel can be deswollen by an external pressure (23).

The extent of reaction and completeness of cross-linking also can be measured. The amount of unreacted monomer in a sample serves as a measure of the extent of monomer conversion in the polymerization reaction. In this technique, the monomer is extracted from the network and quantitated by means of liquid chromatography (24). The completeness of cross-linking can be estimated by measuring the amount of soluble polymer in the product. This is done by extracting the soluble polymer from the network and quantitating it by titration of the carboxylic acid groups (20), or by gravimetry after drying the extract (19). The degree of neutralization of the carboxylic acid groups can be determined by means of a metal-ion specific electrode (25).

Teaching with Superabsorbent Polymers

Chemical Energy and Work

One potential use of superabsorbent polymers is as a muscle-like material. The similarity of cross-linked polymer gels to muscle was recognized years ago by Kuhn and co-workers (26). One property of the gel that is useful in this application is its sensitivity to changes in pH. Polyacrylate superabsorbents swell more in water at high pH than at low pH, and this property can be used as a switch to enable an artificial muscle to expand and contract. The expanding gel can perform useful work, and, therefore, might be used as an artificial finger in robotics (27), for example. The work performed by an expanding gel can be measured in the following way.

Obtain a 12.5-mm diameter glass extraction thimble that has a coarse glass frit in the bottom. Obtain a small plastic disk that fits inside the barrel of the extraction thimble and easily slides within it. One may be cut and machined from a rod of poly(methylmethacrylate). The plastic disk will support brass laboratory weights that will supply various downward forces on the swelling gel. Carefully weigh 120 mg of dry superabsorbent polymer granules into the dry extraction thimble. Tap the thimble gently on a table top to help distribute the polymer granules evenly over the surface of the glass frit. Then carefully

slide the plastic disk down on top of the layer of polymer. Add a small laboratory weight on top of the plastic disk (25–50 g will do nicely). Then place the thimble and its contents into a small, flat-bottomed container, such as a crystallizing dish, and add water to the crystallizing dish until the water level just touches the bottom of the glass frit. (Add a little blue food color to the water to increase the visual impact of the experiment.) The water wicks up through the frit and is absorbed by the superabsorbent polymer. The increasing volume of the gel pushes the piston and brass weight, performing work.

The quantity of work W is determined by measuring the vertical distance d that the disk moves

$$W = Fd$$

where the force F is taken as the product of the mass of the disk and any brass weight, and the acceleration of gravity. The amount of work provided per gram of dry polymer may be calculated from the data.

The work is pressure-volume work and also may be calculated as the pressure P applied to the gel multiplied by the change in volume of the gel. The pressure applied to the gel is given by the force divided by the cross-sectional area of the plastic disk, over which the force is distributed. The volume change of the gel may be determined gravimetrically by measuring the mass of the extraction thimble and its contents both before and after the liquid absorption process, calculating the change in mass and dividing it by the density of the absorbed liquid. If the change in volume per gram of dry polymer is calculated, the work per gram of polymer may be compared to its value as determined by the gel-height method described above. Values for the work per gram of polymer may be measured using different weights and compared. In order to observe changes in gel volume with changing pH, buffer solutions may be poured into the top of the extraction thimble so that they trickle down through the gel. The gel will expand and contract in response to the change in its extent of neutralization. In addition, the gel will expand or contract as salt solutions of various ionic strength are passed through it. This is caused by electrostatic screening of the fixed charges along the contour of the polymer chain.

Size-Exclusion Chromatography and Beer's Absorption Law

Another interesting phenomenon of superabsorbent polymers is their ability to absorb or reject solutes depending on the size of the solute. Here we will compare the absorption of water, containing a macromolecular solute, into the pores of paper and superabsorbent polymer. The porous nature of paper is due to the entanglement of macroscopic fibers of cellulose and the porous nature of superabsorbent polymer is due to the entanglement and cross-linking of the molecular strands of the polymer. Therefore, the pores of the two materials have different characteristic sizes. As a result, solutes of different size can diffuse into the pores. The following experiment demonstrates the different pore sizes of paper and superabsorbent polymer in a very visual manner.

Make up a solution of 0.05% blue dextran (Sigma Chemical) in a previously prepared solution of 0.9% sodium chloride in water. Blue dextran is a high molecular weight polymer that contains a covalently bonded dye. Suspend a strip of filter paper from a hook and dip the lower end into the solution of blue dextran. Let the paper absorb the solution for 5–10 min and note the distance that the liquid has moved. Also observe if the paper is wetted beyond the blue front. No difference should be apparent. Both the water molecules and the macromolecules of dextran diffuse into the large pores of paper. A very different result is obtained with superabsorbent polymer.

Add about 0.5 g of superabsorbent polymer to 60 g of the blue dextran solution prepared above, and let the mixture stand for 60 min. Stir the slurry of gel particles, and let them settle. The layer of gel particles is lighter in color and the liquid phase is darker blue. The particles of superabsorbent polymer absorb the water and salt in the solution, but exclude the macromolecules of dextran because the pores are larger than water or salt but smaller than the dextran molecules. Therefore, the blue polymer becomes more concentrated in the liquid phase. The slurry may be poured into a coarse screen filter to separate the gel from the blue supernatant. The small amount of blue color remaining on the gel can be washed off with a little salt solution. If the change in concentration of the blue-colored material is measured precisely, then the amount of liquid that was absorbed by the superabsorbent polymer can be calculated. The change in concentration of blue-colored material may be found by measuring the intensity of light (617 nm wavelength) transmitted through the blue solution, using a spectrophotometer. By Beer's absorption law, the intensity is proportional to the concentration. The intensity of light passing through the solution of blue polymer is measured both before and after the swelling process, and these values are compared to the intensity of light passing through solutions of known concentration of blue dextran. This technique is the basis of a method to determine accurately the swelling capacity of commercial superabsorbent polymers (20).

Students also could be encouraged to find out how much superabsorbent polymer swells in pure water, or in saline solutions of varying concentration of salt or in saline solutions of salts other than sodium chloride (calcium chloride, for example). In addition, the swelling of these ionic polymers might be studied over a range of pH to see how the extent of ionization of the polymer affects its swelling character. The rate of swelling of superabsorbent polymers in various aqueous solutions also can be interesting. Students could study diffusion phenomena by measuring the dependence of swelling rate on the size of the particles and on the temperature of the liquid. Experiments and observations like these are why superabsorbent polymers have fascinated generations of chemists, engineers, and physicists. In addition, over the past 10 years they have become a common material used by millions of people every day.

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